

TAT-02-F-05480

**AIR AND WIPE SAMPLING PLAN  
SYNKOTE SITE  
ELMWOOD PARK, BERGEN COUNTY, NJ**

September 1989

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## APPENDICES

Appendix A: Wipe Sampling Protocol

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NIOSH 1003

NIOSH 1500

NIOSH 1501

AIR AND WIPE SAMPLING PLAN  
SYNKOTE SITE

1. PROJECT NAME: Synkote  
Elmwood Park  
Bergen County, NJ
2. PROJECT REQUESTED BY: Dwayne Harrington  
Response and Prevention Branch
3. DATE REQUESTED: September 18, 1989
4. DATE OF PROJECT INITIATION: September 25, 1989
5. PROJECT OFFICERS: Michael Mentzel, TAT II  
Beverly Lawson, TAT II
6. QUALITY ASSURANCE OFFICER: Chris D'Onofrio, TAT II
7. PROJECT DESCRIPTION:

A. Site Description

The Synkote Paint site is located in Elmwood Park, New Jersey in Bergen County. The site consists of a one story, 10,000 square foot building on a half acre lot. The site is bordered by Van Riper Avenue to the north, railroad tracks to the south and west and a business parking lot to the east. See Figures 1 and 2.

The building's floor and walls are made of concrete masonry. An examination of the building's piping and ceiling in all rooms revealed no obvious asbestos-like material. The roof of the building is in good condition, no leaks were found. An assessment of the inside of the building revealed 3 areas consisting of several offices, a warehouse/storage room and a processing room. A map of the building is shown in Figure 3. The offices are empty, except for some miscellaneous debris. The storage room has approximately 4 open drums of solid debris, probably non-hazardous in nature.

In summary, the site contains a number of chemical hazards which should be immediately addressed. These include the laboratory bench which contains a number of incompatible substances in close proximity to each other, a bulging drum which could detonate at any time and several drums which are presently releasing their contents to the environment in the rear of the site. It is recommended that measures to secure or remove these hazards be implemented.

B. Objective and Scope

The objective of this project is to provide data pertaining to the nature and relative quantity of possible contamination

(unavailable)

on the floor and wall surfaces inside the building. Also, samples of air will be collected to determine the extent, if any, of airborne contamination.

The scope of the project entails collecting samples from several discrete points. The following groups of samples will be taken:

1. Floor wipe samples, approximately 2 locations.
2. Wall wipe samples, approximately 2 locations
3. Air samples, 4 locations

C. Data Usage:

The air and wipe samples will provide information as to the extent of contamination located in the office area of the building. This will aid EPA while deciding the hot, support and clean zones on site.

D. Parameter Table:

1. Wipe Samples (floor and wall)

PARAMETER	# OF SAMPLES	SAMPLE MATRIX	ANALYTICAL MTD. REF.	SAMPLE PRESERVATION	HOLDING TIME	VOLUME
Metals	4	Sediment	See Attachment A	N/A	Stable	4"x4" Gauge

2. Air Samples:

PARAMETER	# OF SAMPLES	SAMPLE MATRIX	ANALYTICAL MTD. REF.	SAMPLE PRESERVATION	HOLDING TIME	VOLUME
Metals	4	Air	NIOSH 7300 Attachment B	N/A	Stable	1250 Liters On Cassette Filter

PARAMETER	# OF SAMPLES	SAMPLE MATRIX	ANALYTICAL MTD. REF.	SAMPLE PRESERVATION	HOLDING TIME	VOLUME
Volatile	4	Air Organic Compounds	NIOSH 1003 1500, 1501 Attachment D,E,F	N/A	14 days	100 Liter Charcoal Sorbent Tube

8. PROJECT FISCAL INFORMATION:

Sampling equipment and manpower shall be provided by the Technical Assistance Team (TAT) in coordination with the U.S. EPA. All man-hours expended by TAT will be charged to TDD # 02-8906-19.

9. PROJECT ORGANIZATION AND RESPONSIBILITY:

The following is a list of key project personnel and their corresponding responsibilities:

Dwayne Harrington USEPA	Project Director
Michael Mentzel, TAT II	Overall Project Coordination Sampling QC
Beverly Lawson, TAT II	Sampling Operations
Chris D'Onofrio, TAT II	Laboratory Coordination & QC

10. Wipe Sampling:

Wipe sampling methods will follow the procedures outlined in the guidance document included as Attachment A. Four discrete points will be sampled. One blank will also be submitted for analysis. Deionized water will be used as a solvent for the metal samples.

B. Air Sampling:

Air sampling for organic compounds will be conducted in accordance with a modified version of NIOSH Method. Due to the low concentrations of contaminants suspected, a volume of 100 liters will be collected for analysis of all organic constituent in the samples. Analyses of samples collected under Methods Numbers 1003, 1500 and 1501 will be combined from a single charcoal sample tube collected at each location. Samples collected for metals will adhere to the NIOSH Method Procedures Numbers 7300 (see Attachment B). In addition to the sample from each of the four locations shown in Figure 2, 3, three media blanks, one blind duplicate, one MS/MSD, and one field blank will be provided to the lab for QA/QC. All important data pertaining to the air samples will be recorded on the data sheet included as Figure 3.4.

11. 10. SAMPLE CUSTODY PROCEDURES:

Each sample must be accurately and completely identified. It is important that any label be moisture resistant and able to withstand field conditions. Sample containers will be labeled prior to sample collection. The information on each label should include the following, but is not limited to:

- i. Date of collection
- ii. Site name
- iii. Sample identity/location
- iv. Analysis requested

EPA Chain-of-Custody will be filled out and maintained throughout the entire site activities as per TAT SOP on sample handling,

Sampling Container Contract specifications, and EPA Laboratories SOP. The Chain-of-Custody form to be used lists the following information:

- i. Project name;
- ii. Sample number;
- iii. Number of sample containers;
- iv. Description of samples including specific location of sample collection;
- v. Identity of person collecting the sample;
- vi. Date and time of sample collection;
- vii. Date and time of custody transfer to laboratory (if the sample was collected by a person other than laboratory personnel);
- viii. Identity of person accepting custody (if the sample was collected by a person other than laboratory personnel);
- ix. Identity of the lab performing the analyses.

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11. DOCUMENTATION, DATA REDUCTION AND REPORTING:

Field data will be entered into a bound notebook. Field notebooks, field data sheets, Chain-of-Custody forms, and laboratory analyses reports will be filed and stored per the TAT Document Control System.

17 12. QUALITY ASSURANCE AND DATA REPORTING:

QA/QC to be furnished by the contracted laboratory in performance of the analysis will (at a minimum consist of the following measures to ensure accurate data:

1. One field blank for each type of equipment per day will be shipped to the laboratory. These blanks, are to be prepared prior to the sampling events on each day and analyzed in order to ensure that no contamination has occurred during sampling.
2. Three media blanks for the air sampling and one trip blank for the water will be provided daily to ensure the integrity of the samples.
3. A blind duplicate of each matrix will be submitted for every 20 samples to check the analytical precision. Results will be documented and submitted in the written report.
4. Matrix spike and matrix spike duplicate analysis will also be performed on one sample for every 20 samples of each matrix. Triple volume will be collected.
5. The contracted laboratory will also furnish the following deliverables as warranted:

- a) GC/MS tuning and calibration standards;
- b) Copies of all spectral data obtained during performance of analysis. Copies should be signed by the analyst and checked by the Laboratory Manager;
- c) Data System Printout (quantitation report or legible facsimile (GC/MS));
- d) Manual work sheets;
- e) Identification and explanation of any analytical modifications used that differ from USEPA protocol.

All results are to be completed and a written report submitted by the lab to the TAT QC officer within two (2) weeks of the Validated Time of Sample Receipt (VTSR).

14  
13. DATE VALIDATION:

All steps of data generation and handling will be evaluated by the Project Officer and the Quality Assurance Officer for compliance with the specified requirements. ESD-MMB will perform data validation using current protocol.

14. SYSTEM AUDIT:

The Quality Control Officer will observe the sampling operations and subsequent analytical data to assure that the QA/QC project plan has been followed.

15. CORRECTIVE ACTION:

All provisions will be taken in the field and laboratory to ensure that any problems that may develop will be dealt with as quickly as possible. This will be done to ensure the continuity of the sampling program. Any deviations from this sampling plan will be noted in the final report.

16. REPORTS:

Laboratory results and all requested QA/QC information will be submitted to EPA upon completion of sample analyses. Sampling reports will be issued after receipt of laboratory results.



**FIGURE 1, 2, 3, 4**

**APPENDIX A**  
**WIPE SAMPLING PROTOCOLS**

**APPENDIX B**  
**NIOSH METHODS**



**WESTON**

SPILL PREVENTION &  
EMERGENCY RESPONSE DIVISION

In Association with ICF Technology Inc., G. Johnson & Associates,  
Inc., Resource Applications, Inc., Geo Resource Consultants, Inc.,  
and Environmental Toxicology International, Inc.

EPA PM

D. Harrington

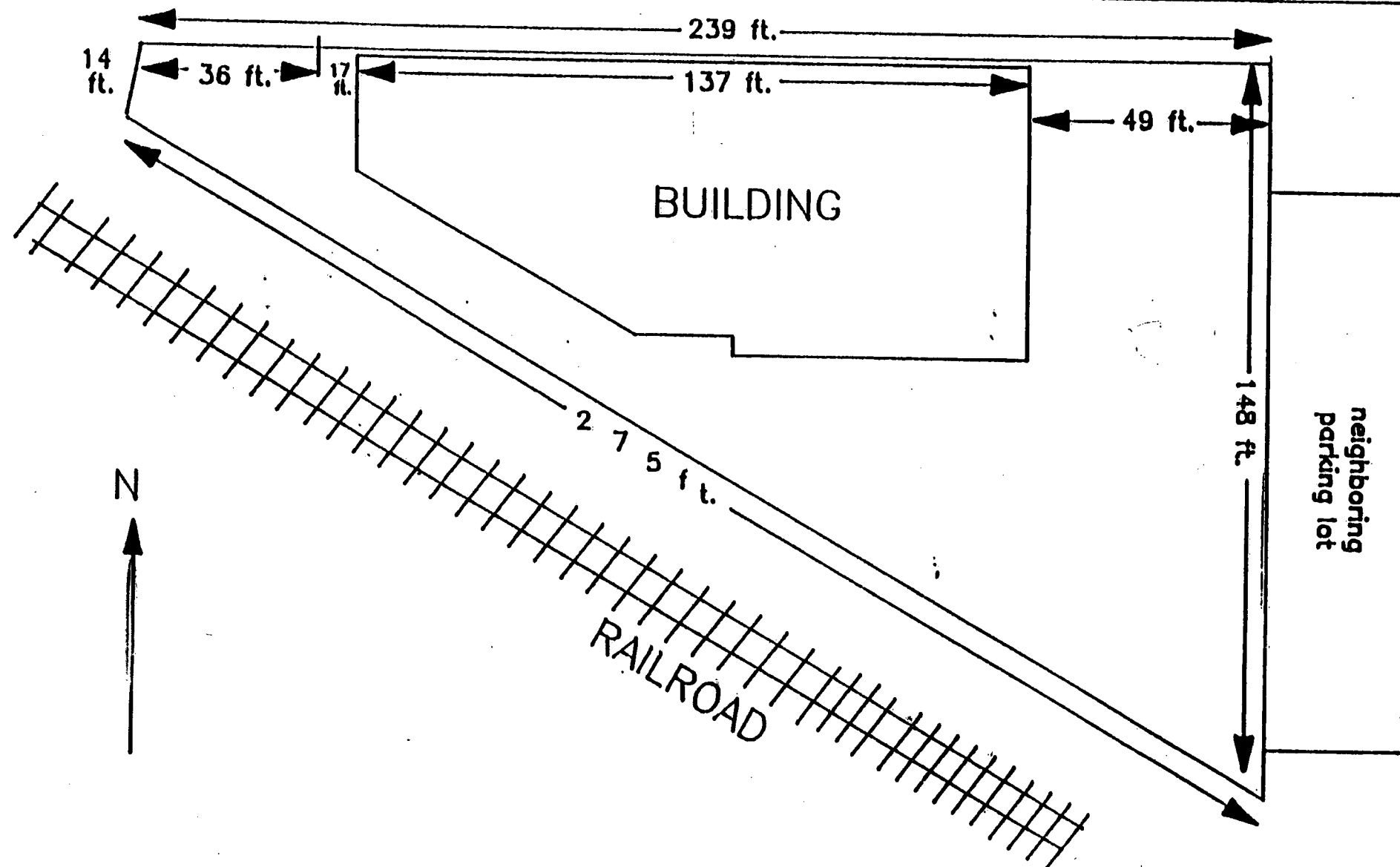
TAT PM

D. Belyung

Figure 1  
Site Location

Scale = 3" = 1 mile

# VAN RIPER AVENUE



SPILL PREVENTION &  
EMERGENCY RESPONSE DIVISION

In Association with ICF Technology Inc., C.C. Johnson & Associates, Inc., Resource Applications, Inc., Geo/Resource Consultants, Inc., and Environmental Toxicology International, Inc.

EPA PM

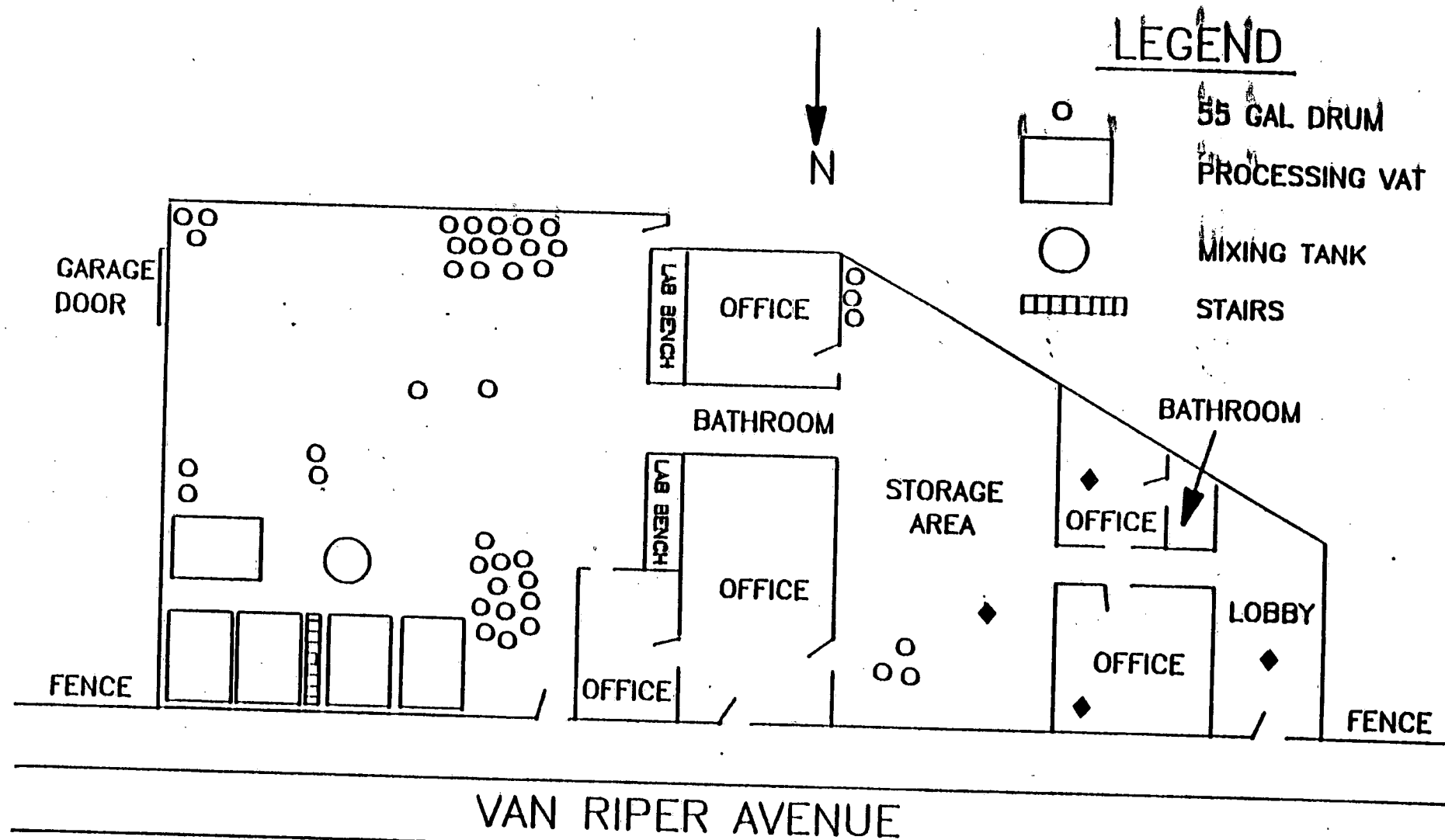
D. Harrington

TAT PM

D. Belyung

Figure 2  
Site Map

Scale = 1" = 30 ft.



◆ = SAMPLE LOCATION POINTS

◆ (BACKGROUND)

**WESTON**  
ENGINEERING CONSULTANTS

SPILL PREVENTION &  
EMERGENCY RESPONSE DIVISION

In Association with ICF Technology Inc., C.C. Johnson & Associates, Inc., Resource Applications, Inc., Geo/Resource Consultants, Inc., and Environmental Toxicology International, Inc.

EPA PM  
D. Harrington

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Figure 3  
Building Interior

Drawing not to Scale

DATE \_\_\_\_\_

SITE NAME \_\_\_\_\_

TEMPERATURE (°F avg.) \_\_\_\_\_

SAMPLED BY \_\_\_\_\_

HUMIDITY (%) \_\_\_\_\_

WIND DIRECTION \_\_\_\_\_

BAROMETRIC PRESSURE \_\_\_\_\_

SAMPLE NO.	LOCATION	PUMP ID NO.	START TIME	START READING	STOP TIME	STOP READING

COMMENTS (RAIN, DAMAGED PUMP, ETC.) \_\_\_\_\_

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION II

DATE:

SUBJECT: Wipe Sample Methodology

FROM: Lisa Gatton, Quality Assurance Chemist  
Monitoring Management Branch

John Birri, Quality Assurance Chemist  
Monitoring Management Branch

TO: Darvene Adams, Environmental Scientist  
Surveillance and Monitoring Branch

The procedures for taking wipe samples as presented in Guide for Decontaminating Buildings, Structures, and Equipment at Superfund Sites, EPA-600-2-85-028, March 1985, and in the NUS Operating Guidelines Manual, can be combined and modified into the following procedure for collecting wipe samples.

1. Materials Needed:

- a. cotton swabs, solvent rinsed and completely air dried
  - b. acetone, pesticide grade
  - c. hexane, pesticide grade
  - d. deionized water
  - e. stainless steel clamps or plastic clamps (only for taking metals samples)
  - f. appropriate sample bottles
2. A square area, of a size sufficient to give the required amount of sample, for each fraction as provided in the analytical methodology to be used, should be marked off. This may require taking cotton swabs and a balance into the field, wiping a certain area and weighing the swab before and after to determine how much area should be wiped to give the required weight of sample.
  3. Swabs for semi-volatile, pesticide and PCB samples should be moistened in a 1:4 acetone/hexane mixture. Swabs for volatile organic samples should be moistened with hexane alone, and those for metals with deionized water. While holding the swab in a clean, metal clamp, moistened the cotton swab with the appropriate solution.
  4. While still holding the cotton swab in the clamp, wipe the sampling area back and forth repeatedly, applying moderate pressure. Wipe the entire area so that all the sample material is picked up.
  5. Place the used swab in the appropriate sample container and seal.
  6. Clean the clamps between each sample with both solvent and 10% HCl.
  7. As a blank, moisten a clean swab with the solvent or water (for each collection medium), place it in a separate jar, and submit it with the other samples.
  8. When samples are submitted for analysis, the laboratory should be told to rinse the sample jars with solvent or 10% HCl when transferring sample to the extraction glassware.



## WIPE SAMPLING PROTOCOL

### I. PURPOSE

Wipe samples will be taken to assess the presence of contaminants on the floor. The major objectives for the wipe samples is to establish whether or not a contaminant is present.

### II. SCOPE

This procedure is applicable to the collection of wipe samples for determining contaminant levels on hard surfaces such as floors, walls and equipment. Wipes are an effective means for collecting a specimen of ambient constituents deposited or settled out on surfaces as a result of some contaminant-releasing incident. Surface areas of personnel contact or those areas associated with air handling systems are highly desirable locations to sample. A thorough understanding of all factors contributing to the condition of contamination, possible sources and the intended use of the data must be taken into account in making appropriate decisions concerning sample location.

When the surface contaminant is in a phase other than particulate (e.g. mist, oil layer, etc.), an appropriate solvent must be used to remove the contamination. For metals a 10% nitric acid solution will be used.

### III. PROCEDURES

#### A. Preparation

1. Prepare wipe collection pads by placing 4 x 4 inch, 100% cotton sterile gauze pads into the wide mouth reservoir jar with gloved hands. Saturate the pads with the nitric acid immediately prior to collecting the sample.
2. Begin the sampling procedure by collecting a field blank by wiping a pair of disposable gloves with a prepared gauze pad. The field blank will determine if specific analytical interferences may be present in either the sorbent pads, solvent or the gloves. This procedure is repeated at a frequency of 5% of samples collected (1 per 20 samples).
3. Specifically locate and measure the area to be sampled and mark it with pencil or a non-interfering tape (e.g., masking tape) or utilize a pre-measured, decontaminated template.
4. Put on a new pair of gloves and press the sampling pad within the designated sample area. With straight, even strokes, draw the pad across the area, slightly overlapping each stroke. Change the wiping direction with a clean pad

and repeat the pattern until confident that all of the surface contaminant has been removed.

5. As each wipe pad is used, place it in the appropriate pre-labeled sample container. When all pads for a sample have been completed, apply custody tape at the closure area of the container.

6. Prepare sample documentation and label the sample area.

B. Location: The sample point can be selected prior to arrival on-site based on previous site visits, maps, etc., or immediately prior to collection based on observations.

Samples will be of a worst-case contamination sample type. This is to determine the area of highest contamination probability.

C. Wipe Area:

A 2500 cm<sup>2</sup> (0.25 m<sup>2</sup>) area will be appropriate for most wipe sample applications. The actual area to be wiped is largely determined by available analytical method sensitivity for target parameters and the target concentration(s) that define allowable exposure levels.

If a single surface area does not provide sufficient area for sample representation, smaller areas from the same general location may be sampled and composited to form one sample for analysis.

D. Solvent Selection

When sampling for particulates, the wipe efficiency is improved by saturating the pad with solvent. While solubility is not the concern in this case, the presence of moisture on the wipe encourages the particulates to cling to the gauze.

#### IV. EQUIPMENT AND SUPPLIES

- \*Nitric acid 10%
- \*4 x 4" 100% cotton sterile gauze pads individually wrapped
- \*wide mouth glass jar with teflon lined lid for solvent/pad reservoir
- \*Appropriately sized sample bottles and screw caps with teflon liners
- \*Custody tape - wide/narrow
- \*Disposable gloves (appropriate to particular situation)
- \*Unique pre-printed sample number label tape
- \*"zip-lock" bags - quart and gallon sizes
- \*Aluminum foil
- \*Masking tape

- \*Duct tape
- \*Metric tape measure
- \*Black indelible pen
- \*Black ball point pen
- \*Black felt tip pen
- \*Field log book
- \*Chain-of-custody forms
- \*Request For Analysis forms
- \*Shipping containers
- \*Shipping labels
- \*Trash bags
- \*Flashlight with extra batteries
- \*Mobile radios, if applicable, with chargers
- \*Paper towels
- \*25', 100' tape measures
- \*Pencils

V.

REFERENCES

Sampling For Surface Contamination. Industrial Hygiene Technical Manual No. 680, May 24, 1984. U.S. Department of Labor: OSHA, p. VIII-1.

# ELEMENTS (ICP)

M.W.: Table 1

METHOD: 7300

ISSUED: 2/15/84

OSHA/NIOSH/ACGIH: Table 1

PROPERTIES: Table 1

ELEMENTS: aluminum	cobalt	manganese	silver	tungsten
arsenic	copper	molybdenum	sodium	vanadium
beryllium	iron	nickel	tellurium	yttrium
cadmium	lead	phosphorus	thallium	zinc
calcium	lithium	platinum	tin	zirconium
chromium	magnesium	selenium	titanium	

SYNONYMS: vary depending upon the compound.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (0.8- $\mu$ m, cellulose ester membrane)	! TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ! ATOMIC EMISSION SPECTROSCOPY
FLOW RATE: 1 to 4 L/min	! ANALYTE: elements above
VOL-MIN: Table 1 -MAX: Table 1	! WASHING REAGENTS: conc. HNO <sub>3</sub> , 4 mL; ! and conc. HClO <sub>4</sub> , 1 mL
SHIPMENT: routine	! CONDITIONS: room temperature, 30 min; ! 150 °C to near dryness
SAMPLE STABILITY: stable	! FINAL SOLUTION: 4% HNO <sub>3</sub> , 1% HClO <sub>4</sub> , 10 mL
BLANKS: 2 to 10 field blanks per set	! WAVELENGTH: depends upon element; Table 2
	! BACKGROUND CORRECTION: spectral wavelength shift
	! CALIBRATION: elements in 4% HNO <sub>3</sub> , 1% HClO <sub>4</sub>
	! RANGE: 2.5 to 1000 $\mu$ g per sample [1]
	! ESTIMATED LOD: 1 $\mu$ g per sample [1]
	! PRECISION (s <sub>p</sub> ): Table 2

## ACCURACY

RANGE STUDIED: not studied

BIAS: none identified

OVERALL PRECISION (s<sub>p</sub>): not evaluated

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m<sup>3</sup> for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with this ashing procedure.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1,2].

OTHER METHODS: This method replaces P&CAM 351 [2] for trace elements. Atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements.

## REAGENTS:

1. Nitric acid, conc.
2. Perchloric acid, conc.\*
3. Ashing acid: 4:1 (v/v)  $\text{HNO}_3$ : $\text{HClO}_4$ .  
Mix 4 volumes conc.  $\text{HNO}_3$  with  
1 volume conc.  $\text{HClO}_4$ .
4. Calibration stock solutions,  
1000  $\mu\text{g/mL}$ . Commercially available,  
or prepared per instrument  
manufacturer's recommendation (see  
step 12).
5. Dilution acid, 4%  $\text{HNO}_3$ , 1%  $\text{HClO}_4$ .  
Add 50 mL ashing acid to 600 mL  
water; dilute to 1 L.
6. Argon.
7. Distilled, deionized water.

\*See Special Precautions.

## EQUIPMENT:

1. Sampler: cellulose ester membrane filter,  
0.8- $\mu\text{m}$  pore size, 37-mm diameter; in cassette  
filter holder.
2. Personal sampling pump, 1 to 4 L/min, with  
flexible connecting tubing.
3. Inductively coupled plasma-atomic emission  
spectrometer, equipped as specified by the  
manufacturer for analysis of elements of interest.
4. Regulator, two-stage, for argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with  
watchglass covers.\*
6. Volumetric flasks, 10- and 100- mL.\*
7. Assorted volumetric pipets as needed.\*
8. Hotplate, surface temperature 150  $^{\circ}\text{C}$ .

\*Clean all glassware with conc. nitric acid and  
rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: Perform all perchloric acid digestions in a perchloric acid hood.

## SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of  
200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of  
approximately 2 mg total dust.

## SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature.  
NOTE: Start a reagent blank at this step.
5. Heat on hotplate (120  $^{\circ}\text{C}$ ) until ca. 0.5 mL remains.  
NOTE: Some species of Li, Mn, Mo, Sn, W, and Zr will not be completely solubilized by this  
procedure. Alternative solubilization techniques for most of these elements can be  
found elsewhere [2,3,4,5,6,7].
6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
7. Remove watchglass and rinse into the beaker with distilled water.
8. Increase the temperature to 150  $^{\circ}\text{C}$  and take the sample to dryness.
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 10-mL volumetric flasks.
11. Dilute to volume with dilution acid.

## CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.  
NOTE: Typically, an acid blank and 10  $\mu\text{g/mL}$  multielement working standards are used. The  
following multielement combinations are chemically compatible in 4%  $\text{HNO}_3$ /1%  $\text{HClO}_4$ :
  - a. Ag, Ca, Co, Mn, Pb, V, Zn;
  - b. Al, Be, Cd, La, Li, Ni, Ti;
  - c. As, B, Ba, Mg, Mo, P, Sn;

- d. Cu, Fe, Na, Pt, Sr, Te, Y;
- e. Cr, K, Sb, Se, Ti, Zr; and
- f. Si, W (distilled water only)

- 13. Analyze a standard for every ten samples.
- 14. Check recoveries with at least two spiked media blanks per ten samples.

**MEASUREMENT:**

- 15. Set spectrometer to conditions specified by manufacturer.
- 16. Analyze standards and samples.

NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

**CALCULATIONS:**

- 17. Obtain the solution concentrations for the sample,  $C_s$  ( $\mu\text{g/mL}$ ), and the average media blank,  $C_b$  ( $\mu\text{g/mL}$ ), from the instrument.
- 18. Using the solution volumes of sample,  $V_s$  (mL), and media blank,  $V_b$  (mL), calculate the concentration,  $C$  ( $\text{mg/m}^3$ ), of each element in the air volume sampled,  $V$  (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

**EVALUATION OF METHOD:**

Method P&CAM 351 was evaluated in 1981 [1,2]. The precision and recovery data were determined at 2.5 and 1000  $\mu\text{g}$  of each element per sample on spiked filters. The precision and recovery data, instrumental detection limits, sensitivity, and analytical wavelengths are listed in Table 2. The values in Table 2 were determined with a Jarrell-Ash Model 1160 ICP operated according to manufacturer's instructions.

**REFERENCES:**

- [1] Hull, R.D. "Multielement Analysis of Industrial Hygiene Samples," NIOSH Internal Report, presented at the American Industrial Hygiene Conference, Portland, Oregon (May 1981).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
- [3] Ibid, S341 (Lead).
- [4] Ibid, V. 2, S5 (Manganese), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [5] Ibid, V. 4, P&CAM 271 (Tungsten), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [6] Ibid, V. 5, P&CAM 173 (Metals by Atomic Absorption), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
- [7] Ibid, V. 3, S183 (Tin), S185 (Zirconium), and S376 (Molybdenum), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY: R. DeLon Hull and Mark Millson, NIOSH/DPSE.

Table 1. Properties and sampling volumes.

Element (Symbol)	Properties		Permissible Exposure Limits, mg/m <sup>3</sup> TWA OSHA/NIOSH/ACGIH	Air Volume @ OSHA, L	
	Atomic Weight	MP, °C		MIN	MAX
Silver (Ag)	107.87	961	0.01/ — / 0.1	250	2000
Aluminum (Al)	26.98	660	— / — / 10.	5 (g)	100 (g)
Arsenic (As)	74.92	817*	0.5/C 0.002/ 0.2	5	2000
Beryllium (Be)	9.01	1278	0.002/ 0.0005/ 0.002	1250	2000
Calcium (Ca)	40.08	842	5 (b)/ — / 2 (b)	5	200
Cadmium (Cd)	112.40	321	0.2/ 0.04/ 0.05	13	2000
Cobalt (Co)	58.93	1495	0.1/ — / 0.1	25	2000
Chromium (Cr)	52.00	1890	1.0 (c)/ 0.025/ 0.5 (c)	5	1000
Copper (Cu)	63.54	1083	1.0/ — / 1.0	5	1000
Iron (Fe)	55.85	1535	10 (b)/ — / 5 (b)	5	100
Lithium (Li)	6.94	179	0.025 (d)/ — / 0.025 (d)	100	2000
Magnesium (Mg)	24.31	651	15 (b)/ — / 10 (b)	5	67
Manganese (Mn)	54.94	1244	C 5/ — / C 5	5	200
Molybdenum (Mo)	95.94	651	15 (e)/ — / 10 (e)	5	67
Sodium (Na)	22.99	98	2 (f)/ C 2 (f)/ C 2 (f)	13	2000
Nickel (Ni)	58.71	1453	1/ 0.015/ 1 (c)	5	1000
Phosphorus (P)	30.97	44	— / — / 0.1	25 (g)	2000 (g)
Lead (Pb)	207.19	328	0.05/ 0.1/ 0.15	50	2000
Platinum (Pt)	195.09	1769	0.002 (a)/ — / 1 (c)	1250	2000
Selenium (Se)	78.96	217	0.2/ — / —	13	2000
Tin (Sn)	118.69	232	2/ — / 2 (c)	5	500
Tellurium (Te)	127.60	450	0.1/ — / 0.1	25	2000
Titanium (Ti)	47.90	1675	— / — / 10 (b)	5	100
Thallium (Tl)	204.37	304	0.1 (a)/ — / 0.1 (a)	25	2000
Vanadium (V)	50.94	1890	C 0.5/ 1 (c)/ 0.05 (V <sub>2</sub> O <sub>5</sub> )	5	2000
Tungsten (W)	183.85	3410	— / 5 (e)/ 5 (e)	5 (g)	200 (g)
Yttrium (Y)	88.91	1495	1/ — / 1	5	1000
Zinc (Zn)	65.37	419	5 (b)/ 5 (b)/ 5 (b)	5	200
Zirconium (Zr)	91.22	1852	5/ — / 5	5	200

(a) soluble

(b) oxide

(c) metal

(d) hydride

(e) insoluble

(f) hydroxide

(g) at the ACGIH TLV

Table 2. Measurement procedures and data (a).

Element	Wavelength (nm)	Instrumental LOD (ng/mL)	Sensitivity (Intensity/ µg/mL)	Recovery (%)		Precision (s <sub>p</sub> ) (N = 3)	
				@ 2.5 µg/ filter (b)	@ 1000 µg/ filter	@ 2.5 µg/ filter	@ 1000 µg/ filter
Ag	328.3	26	0.65	111	91	0.02	0.075
Al	308.2	14	0.23	93	100	0.092	0.023
As	193.7	13	0.57	103	99	0.062	0.026
Be	313.0	1.5	1.29	107	90	0.040	0.034
Ca	315.9	10	0.49	99	95	0.036	0.014
Cd	226.5	1.6	0.83	107	99	0.032	0.020
Co	231.2	7.4	0.38	101	95	0.040	0.005
Cr	205.6	1.3	0.50	98	106	0.053	0.016
Cu	324.8	2.1	0.72	98	99	0.036	0.022
Fe	259.9	3.9	0.13	94	97	0.068	0.016
Li	670.8	2.8	0.48	89	95	0.171	0.043
Mg	279.6	24	0.22	105	106	0.084	0.027
Mn	257.6	0.4	0.74	84	93	0.062	0.035
Mo	281.6	7.0	0.18	94	88	0.023	0.049
Na	589.0	10	0.76	(c)	101	(c)	0.045
Ni	231.6	3.4	0.41	105	97	0.027	0.020
P	214.9	22	0.17	(c)	91	(c)	0.056
Pb	220.4	17	0.42	105	95	0.060	0.011
Pt	203.7	15	0.69	106	91	0.041	0.075
Se	190.6	21	0.28	105	97	0.068	0.049
Sn	190.0	64	0.49	74	67	0.33	0.16
Te	214.3	29	0.41	102	94	0.050	0.063
Ti	334.9	1.2	0.55	96	108	0.051	0.029
Tl	190.9	17	0.22	103	99	0.043	0.017
V	310.2	3.2	0.88	99	94	0.043	0.014
W	207.9	13	2.58	35	23	0.053	0.60
Y	371.0	0.8	2.35	99	100	0.015	0.013
Zn	213.9	0.6	0.60	101	94	0.013	0.013
Zr	339.2	1.9	0.88	75	98	0.049	0.008

(a) Values reported were obtained with a Jarrell-Ash Model 1160 ICP; performance may vary with instrument and should be independently verified.

(b) 2.5 µg/filter corresponds to 5 µg/m<sup>3</sup> for a 500-L air sample.

(c) Blank levels too high to make accurate determinations



FORMULA: Table 1

## HYDROCARBONS, BP 36 – 126 °C

M.W.: Table 1

METHOD: 1500

ISSUED: 2/15/84

OSHA, NIOSH, ACGIH: Table 2

PROPERTIES: Table 1

COMPOUNDS:	benzene	n-heptane	n-octane
(Synonyms	cyclohexane	n-hexane	n-pentane
in Table 1)	cyclohexene	methylcyclohexane	toluene

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	! TECHNIQUE: GAS CHROMATOGRAPHY, FID ! ! ANALYTES: hydrocarbons listed above !
FLOW RATE, VOLUME: Table 3	! DESORPTION: 1 mL CS <sub>2</sub> ; stand 30 min !
SHIPMENT: no special precautions	! INJECTION VOLUME: 5 µL !
SAMPLE STABILITY: at least 2 weeks	! TEMPERATURE-INJECTION: 250 °C ! -DETECTOR: 250 °C ! -COLUMN: see step 11 !
BLANKS: 2 to 10 field blanks per set	!
BULK SAMPLE: desirable, 1 to 10 mL; ship in separate containers from samples	! CARRIER GAS: N <sub>2</sub> or He, 25 mL/min ! ! COLUMN: glass, 3.0 m x 2 mm, 20% SP-2100 on 80/100 mesh Supelcoport !
ACCURACY	! CALIBRATION: analytes in CS <sub>2</sub> !
RANGE STUDIED, BIAS and OVERALL PRECISION (s <sub>p</sub> ): Table 3	! RANGE AND PRECISION (s <sub>p</sub> ): Table 4 ! ! ESTIMATED LOD: 0.001 to 0.01 mg per sample with capillary column [1] !

**APPLICABILITY:** This method is intended for determining the OSHA-regulated hydrocarbons included within the boiling point range of n-pentane through n-octane. It may be used for simultaneous measurements; however, interactions between analytes may reduce breakthrough volumes and change desorption efficiencies.

**INTERFERENCES:** At high humidity, breakthrough volumes may be reduced by as much as 50%. Other volatile organic solvents, e.g., alcohols, ketones, ethers, and halogenated hydrocarbons, are likely interferences. If interference is suspected, use a more polar column or change column temperature.

**OTHER METHODS:** This method is based on and supercedes Methods P&CAM 127, benzene and toluene [2]; S28, cyclohexane [3]; S82, cyclohexene [3]; S89, heptane [3]; S90, hexane [3]; S94, methylcyclohexane [3]; S311, benzene [4]; S343, toluene [4]; S378, octane [4]; and S379, pentane [4]. For benzene or toluene in complex mixture of alkanes (≤C<sub>10</sub>), Method 1501 (aromatic hydrocarbons) is more selective.

## REAGENTS:

1. Eluent: Carbon disulfide\*, chromatographic quality with (optional) suitable internal standard.
2. Analytes, reagent grade.\*
3. Nitrogen or helium, purified.
4. Hydrogen, prepurified.
5. Air, filtered.

\*See Special Precautions.

## EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6 mm OD, 4 mm ID, flame-sealed ends, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg, back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section, and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (page 1500-1).
4. Vials, glass, 1-mL, with PTFE-lined caps.
5. Pipet, 1-mL, with pipet bulb.
6. Syringes, 5-, 10-, 25- and 100- $\mu$ L.
7. Volumetric flasks, 10-mL

**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and extremely flammable (flash point = -30 °C); benzene is a suspect carcinogen. Prepare samples and standards in a well-ventilated hood.

## SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min (0.01 to 0.05 L/min for n-pentane) for a total sample size as shown in Table 3.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

## SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial immediately.
7. Allow to stand at least 30 min with occasional agitation.

## CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the appropriate range (ca. 0.01 to 10 mg analyte per sample; see Table 4).
  - a. Add known amounts of analyte to eluent in 10-mL volumetric flasks and dilute to the mark.
  - b. Analyze together with samples and blanks (steps 11, 12 and 13).
  - c. Prepare calibration graph (peak area of analyte vs. mg analyte).
9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
  - a. Remove and discard back sorbent section of a media blank sampler.
  - b. Inject a known amount of analyte directly onto front sorbent section with a microliter syringe.

- c. Cap the tube. Allow to stand overnight.
- d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11, 12 and 13).
- e. Prepare a graph of DE vs. mg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control. Check for possible contamination during shipment of field samples by comparing results from field blanks and media blanks.

## MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1500-1. Select appropriate column temperature:

Substance	Approximate Retention Time (min), at Indicated Column Temperature			
	40 °C	70 °C	100 °C	Programmed <sup>a</sup>
n-pentane	2.2	1.2		1.8
solvent (CS <sub>2</sub> )	3.0	1.6		2.4
n-hexane	5.1	2.2		3.5
benzene <sup>b</sup>	7.7	3.2		4.5
cyclohexane <sup>b</sup>	8.4	3.4		4.7
cyclohexene	9.5	3.8		4.9
n-heptane	12	4.3		5.4
methylcyclohexane	14	5.2	2.2	5.9
toluene	17	6.5	2.6	6.5
n-octane	19	8.7	3.2	7.1

<sup>a</sup>Temperature program: 50 °C for 2 min, then 15 °C/min to 150 °C, 2-min final hold.

<sup>b</sup>Not completely resolved.

NOTE: Alternatively, column and temperature may be taken from Table 4.

12. Inject sample aliquot manually using solvent flush technique or with autosampler.  
NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure peak area.

## CALCULATIONS:

14. Determine the mass, mg (corrected for DE) of analyte found in the sample front (W<sub>f</sub>) and back (W<sub>b</sub>) sorbent sections, and in the average media blank front (B<sub>f</sub>) and back (B<sub>b</sub>) sorbent sections.

NOTE: If W<sub>b</sub> > W<sub>f</sub>/10, report breakthrough and possible sample loss.

15. Calculate concentration, C, of analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

## EVALUATION OF METHOD:






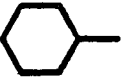


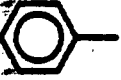
Precisions and biases (Table 3) were determined by analyzing generated atmospheres containing one-half, one, and two times the OSHA standard. Generated concentrations were independently verified. Breakthrough capacities were determined in dry air. Storage stability was not assessed. Measurement precisions (Table 4) were determined by spiking sampling media with amounts corresponding to one-half, one, and two times the OSHA standard for nominal air volumes. Desorption efficiencies for spiked samplers containing only one compound exceeded 75%. Reference [12] provides more specific information.

## REFERENCES:

- [1] User check, UBTI, NIOSH Sequence #4213-L (unpublished, January 31, 1984).
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 127 U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [3] NIOSH Manual of Analytical Methods, 2nd. ed., V. 2, S28, S82, S89, S90, S94, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] NIOSH Manual of Analytical Methods, 2nd. ed., V. 3., S311, S343, S378, S379, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [5] R. D. Driesbach, "Physical Properties of Chemical Compounds"; Advances in Chemistry Series, No. 15; American Chemical Society, Washington (1955).
- [6] R. D. Driesbach, "Physical Properties of Chemical Compounds - II"; Advances in Chemistry Series, No. 22; American Chemical Society, Washington (1959).
- [7] Code of Federal Regulations; Title 29 (Labor), Parts 1900 to 1910; U.S. Government Printing Office, Washington, (1980); 29 CFR 1910.1000.
- [8] Update Criteria and Recommendations for a Revised Benzene Standard, U.S. Department of Health, Education, and Welfare, (August 1976).
- [9] Criteria for a Recommended Standard....Occupational Exposure to Alkanes (C5-C8), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-151 (1977).
- [10] Criteria for a Recommended Standard....Occupational Exposure to Toluene, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11023 (1973).
- [11] TLVs--Threshold Limit Values for Chemical Substances and physical Agents in the Work Environment with Intended Changes for 1983-84, ACGIH, Cincinnati, OH (1983).
- [12] Documentation of the NIOSH Validation Tests, S28, S82, S89, S90, S94, S311, S343, S378, S379, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD REVISED BY: R. Alan Lunsford, Ph.D., and Julie R. Okenfuss; based on results of NIOSH Contract CDC-99-74-45.

Table 1. Synonyms, formula, molecular weight, properties.

Name Synonyms	Structure	Empirical Formula	Molec- ular Weight	Boiling Point (°C)	Vapor Pressure @ 25 °C		Density @ 20 °C (g/mL)
					(mm Hg)	(kPa)	
benzene <sup>a</sup> CAS #71-43-2		C <sub>6</sub> H <sub>6</sub>	78.11	80.1	95.2	12.7	0.879
cyclohexane <sup>a</sup> CAS #110-82-7 hexahydrobenzene hexamethylene		C <sub>6</sub> H <sub>12</sub>	84.16	80.7	97.6	13.0	0.779
cyclohexene <sup>a</sup> CAS #110-83-8 tetrahydrobenzene		C <sub>6</sub> H <sub>10</sub>	82.15	83.0	88.8	11.8	0.811
n-heptane <sup>b</sup> CAS #142-82-5		C <sub>7</sub> H <sub>16</sub>	100.21	98.4	45.8	6.1	0.684
n-hexane <sup>b</sup> CAS #110-54-3		C <sub>6</sub> H <sub>14</sub>	86.18	68.7	151.3	20.2	0.659
methylcyclohexane <sup>a</sup> CAS #108-87-2		C <sub>7</sub> H <sub>14</sub>	98.19	100.9	46.3	6.2	0.769
n-octane <sup>b</sup> CAS #111-65-9		C <sub>8</sub> H <sub>18</sub>	114.23	125.7	14.0	1.9	0.703
n-pentane <sup>b</sup> CAS #109-66-0		C <sub>5</sub> H <sub>12</sub>	72.15	36.1	512.5	68.3	0.626
toluene <sup>a</sup> CAS #108-88-3 methylbenzene		C <sub>7</sub> H <sub>8</sub>	92.14	110.6	28.4	3.8	0.867

<sup>a</sup>Properties from [5].<sup>b</sup>Properties from [6].

Table 2. Permissible exposure limits, ppm [7-11].

Substance	OSHA			NIOSH		ACGIH		mg/m <sup>3</sup> per ppm @ NTP
	TWA	C	Peak	TWA	C	TLV	STEL	
benzene*	10	25	50 <sup>b</sup>	1		10	25	3.19
cyclohexane	300					300	375	3.44
cyclohexene	300					300		3.36
n-heptane	500			85	440	400	500	4.10
n-hexane <sup>a</sup>	500			100	510	50		3.52
methylcyclohexane	500					400	500	4.01
n-octane	500			75	385	300	375	4.67
n-pentane	1000			120	610	600	750	2.95
toluene	200	300	500 <sup>b</sup>	100	200 <sup>c</sup>	100	150 (skin)	3.77

<sup>a</sup>The ACGIH recommendation for other hexane isomers is: TLV 500, STEL 1000.

<sup>b</sup>Maximum duration 10 min in 8 hr.

<sup>c</sup>10-min sample.

\*ACGIH: suspect carcinogen

Table 3. Sampling flowrate<sup>a</sup>, volume, capacity, range, overall bias and precision [2-4, 12].

Substance	Sampling			Breakthrough		Range	Overall	
	Flowrate (L/min)	Volume (L)		Volume at		at	Bias (%)	Precision (s <sub>p</sub> )
		Concentration		(L)	(mg/m³)	VOL-NOM		
		VOL-NOM	VOL-MAX <sup>b</sup>			(mg/m³)		
benzene	≤0.20	2 <sup>c</sup>	30	>45	149.1	41.5 - 165	0.8	0.059
cyclohexane	≤0.20	2.5	5	7.6	1650	510 - 2010	5.4	0.060 <sup>d</sup>
cyclohexene	≤0.20	5	7	10.4	2002	510 - 2030	9.0	0.073
n-heptane	≤0.20	4	4	6.1	4060	968 - 4060	-6.5	0.056
n-hexane	≤0.20	4	4	5.9	3679	877 - 3679	-3.8	0.062
methylcyclohexane	≤0.20	4	4	6.1	3941	940 - 3941	5.5	0.052
n-octane	≤0.20	4	4	6.5	4612	1050 - 4403	-5.2	0.060
n-pentane	≤0.05	2	2	3.1	5640	1476 - 6190	-9.7	0.055
toluene	≤0.20	2 <sup>c</sup>	8	11.9	2294	548 - 2190	3.8	0.052

<sup>a</sup>Minimum recommended flow is 0.01 L/min.

<sup>b</sup>Approximately two-thirds the breakthrough volume.

<sup>c</sup>10-min sample.

<sup>d</sup>Corrected value, calculated from data in [12].

Table 4. Measurement range, precision, and chromatographic conditions [2-4,12].

Substance	Measurement <sup>a</sup>		Carrier		Column Parameters <sup>b</sup>			
	Range	Precision	Gas	Flow	t	Length	Dia-	Packing <sup>c</sup>
	(mg)	(s <sub>r</sub> )		(mL/min)	(°C)	(m)	meter (mm)	
benzene	0.09-0.35	0.036	N <sub>2</sub>	50	115	0.9	3.2	A
cyclohexane	1.3 - 5.3 <sup>d</sup>	0.024	N <sub>2</sub>	50	210	1.2	6.4	B
cyclohexene	2.4 - 9.7 <sup>d</sup>	0.021	N <sub>2</sub>	50	205	1.2	6.4	B
n-heptane	4.08-16.3	0.016	He	30	80	3.0	3.2	C
n-hexane	3.56-14.5	0.014	He	30	52	6.1	3.2	D
methylcyclohexane	3.98-16.1	0.012	He	30	55	6.1	3.2	D
n-octane	4.75-18.9	0.009	He	30	52	6.1	3.2	D
n-pentane	2.98-11.8	0.014	He	30	52	6.1	3.2	D
toluene	1.13-4.51	0.011	N <sub>2</sub>	50	155	0.9	3.2	B

<sup>a</sup>Injection volume, 5.0 µL; desorption volume, 1.0 mL, except cyclohexane and cyclohexene, 0.5 mL.

<sup>b</sup>All columns stainless steel. Diameter is outside dimension.

<sup>c</sup>A, 50/80 mesh Porapak P; B, 50/80 mesh Porapak Q; C, 10% OV-101 on 100/120 mesh Supelcoport; D, 10% FFAP on 80/100 mesh Chromosorb W AW-DMCS.

<sup>d</sup>Corrected value, calculated from data in [12].

FORMULA: Table 1

## HYDROCARBONS, AROMATIC

M.W.: Table 1

METHOD: 1501

ISSUED: 2/15/84

OSHA, NIOSH, ACGIH: Table 2

PROPERTIES: Table 1

COMPOUNDS:	benzene	cumene	$\alpha$ -methylstyrene	styrene	vinyltoluene
(Synonyms in Table 1)	p-tert-butyltoluene	ethylbenzene	naphthalene	toluene	xylene

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE -(coconut shell charcoal, 100 mg/50 mg)	! TECHNIQUE: GAS CHROMATOGRAPHY, FID ! ! ANALYTES: hydrocarbons listed above !
FLOW-RATE, VOLUME: Table 3	! DESCRIPTION: 1 mL CS <sub>2</sub> ; stand 30 min !
SHIPMENT: no special precautions	! INJECTION VOLUME: 5 $\mu$ L !
SAMPLE STABILITY: not determined	! TEMPERATURE-INJECTION: 225 $^{\circ}$ C ! -DETECTOR: 225 $^{\circ}$ C ! -COLUMN: see step 11 !
BLANKS: 2 to 10 field blanks per set	!
BULK SAMPLE: desirable, 1 to 10 mL; ship in separate containers from samples	! CARRIER GAS: N <sub>2</sub> or He, 25 mL/min ! ! COLUMN: glass, 3.0 m x 2 mm, 10% OV-275 on 100/120 mesh Chromosorb W-AW or equivalent !
ACCURACY	! CALIBRATION: analytes in CS <sub>2</sub> ! ! RANGE AND PRECISION (s <sub>r</sub> ): Table 4 ! ! ESTIMATED LOD: 0.001 to 0.01 mg per sample with capillary column [1] !
RANGE STUDIED, BIAS and OVERALL PRECISION (s <sub>r</sub> ): Table 3	

APPLICABILITY: This method is for peak, ceiling and TWA determinations of aromatic hydrocarbons. It may be used for simultaneous measurements, though there is the possibility that interactions between analytes may reduce the breakthrough volumes and change desorption efficiencies.

INTERFERENCES: Use of the recommended column will prevent interference by alkanes ( $\leq$ C<sub>10</sub>). Under conditions of high humidity, the breakthrough volumes may be reduced by as much as 50%. Other volatile organic solvents, e.g., alcohols, ketones, ethers and halogenated hydrocarbons, are possible interferences. If interference is suspected, use a less polar column or change column temperature.

OTHER METHODS: This method is based on and supercedes Methods P&CAM 127, benzene, styrene, toluene and xylene [2]; S311, benzene [4]; S22, p-tert-butyltoluene [3]; S23, cumene [3]; S29, ethylbenzene [3]; S26,  $\alpha$ -methylstyrene [3]; S292, naphthalene [4]; S30, styrene [3]; S343, toluene [4]; S25, vinyltoluene [3]; S318, xylene [4].



## REAGENTS:

1. Eluent: Carbon disulfide\*, chromatographic quality containing (optional) suitable internal standard.
2. Analytes, reagent grade\*
3. Nitrogen or helium, purified
4. Hydrogen, prepurified.
5. Air, filtered.
6. Naphthalene calibration stock solution, 0.40 g/mL in CS<sub>2</sub>.

\*See Special Precautions.

## EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6 mm OD, 4 mm ID, flame-sealed ends, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg, back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pumps, 0.01 to 1 L/min (Table 3), with flexible connecting tubing.
3. Gas chromatograph, FID, integrator, and column (page 1501-1).
4. Vials, glass, 1-mL, with PTFE-lined caps.
5. Pipet, 1-mL, and pipet bulb.
6. Syringes, 5-, 10-, 25- and 100- $\mu$ L.
7. Volumetric flasks, 10-mL.

**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and extremely flammable (flash point = -30 °C); benzene is a suspect carcinogen. Prepare samples and standards in a well-ventilated hood.

## SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min (to 1 L/min for naphthalene or styrene) for a total sample size as shown in Table 3.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

## SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial immediately.
7. Allow to stand at least 30 min with occasional agitation.

## CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the appropriate range (ca. 0.01 to 10 mg analyte per sample; see Table 4).
  - a. Add known amounts of analyte (calibration stock solution for naphthalene) to eluent in 10-mL volumetric flasks and dilute to the mark.
  - b. Analyze together with samples and blanks (steps 11, 12 and 13).
  - c. Prepare calibration graph (peak area of analyte vs. mg analyte).

9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
  - a. Remove and discard back sorbent section of a media blank sampler.
  - b. Inject a known amount of analyte (calibration stock solution for naphthalene) directly onto front sorbent section with a microliter syringe.
  - c. Cap the tube. Allow to stand overnight.
  - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11, 12 and 13).
  - e. Prepare a graph of DE vs. mg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

## MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1501-1. Select appropriate column temperature:

Substance <sup>a</sup>	Approximate Retention Time (min), at Indicated Column Temperature			
	50 °C	100 °C	150 °C	Programmed <sup>b</sup>
benzene	2.5			2.5
toluene	4.3	1.1		4.2
xylene (para)	7.0	1.4		5.2
ethylbenzene	7.0	1.4		5.5
xylene (meta)	7.2	1.5		5.6
cumene	8.3	1.6		6.0
xylene (ortho)	10	1.9		6.5
styrene	16	2.6		7.6
$\alpha$ -methylstyrene		3.2	1.0	8.1
vinyltoluene (meta)		3.8	1.2	8.5
naphthalene		25	4.3	12

<sup>a</sup>Data not available for p-tert-butyltoluene and p-vinyltoluene.

<sup>b</sup>Temperature program: 50 °C for 3 min, then 15 °C/min to 200 °C.

NOTE: Alternatively, column and temperature may be taken from Table 4.

12. Inject sample aliquot manually using solvent flush technique or with autosampler.
 

NOTE: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure peak area.

## CALCULATIONS:

14. Determine the mass, mg (corrected for DE) of analyte found in the sample front ( $W_f$ ) and back ( $W_b$ ) sorbent sections, and in the average media blank front ( $B_f$ ) and back ( $B_b$ ) sorbent sections.

NOTE: If  $W_b > W_f/10$ , report breakthrough and possible sample loss.

15. Calculate concentration, C, of analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

#### EVALUATION OF METHOD:


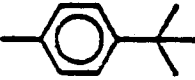
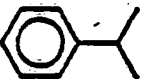
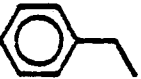
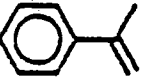

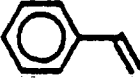
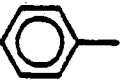
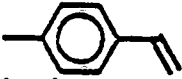

Precisions and biases listed in Table 3 were determined by analyzing generated atmospheres containing one-half, one, and two times the OSHA standard. Generated concentrations were independently verified. Breakthrough capacities were determined in dry air. Storage stability was not assessed. Measurement precisions given in Table 4 were determined by spiking sampling media with amounts corresponding to one-half, one, and two times the OSHA standard for nominal air volumes. Desorption efficiencies for spiked samplers containing only one compound exceeded 75%. Reference [12] provides more specific information.

#### REFERENCES:

- [1] User check, UBTL, NIOSH Sequence #4121-S (unpublished, December 7, 1983).
- [2] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [3] Ibid, V. 2, S22, S23, S25, S26, S29, S30, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] Ibid, V. 3, S292, S311, S318, S343, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [5] R. D. Dreisbach. "Physical Properties of Chemical Compounds"; Advances in Chemistry Series, No. 15; American Chemical Society, Washington (1955).
- [6] Code of Federal Regulations; Title 29 (Labor), Parts 1900 to 1910; U.S. Government Printing Office, Washington (1980); 29 CFR 1910.1000.
- [7] Update Criteria and Recommendations for a Revised Benzene Standard, U.S. Department of Health, Education, and Welfare, (August 1976).
- [8] Criteria for a Recommended Standard....Occupational Exposure to Toluene, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 73-11023 (1973).
- [9] Criteria for a Recommended Standard....Occupational Exposure to Xylene, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 75-168 (1975).
- [10] TLVs - Threshold Limit Values for Chemical Substances and Physical Agents in the Work Environment with Intended Changes for 1983-84, ACGIH, Cincinnati, OH (1983).
- [11] Criteria for a Recommended Standard...Occupational Exposure to Styrene, U.S. Department of Health and Human Services, Publ. (NIOSH) 83-119 (1983).
- [12] Documentation of the NIOSH Validation Tests, S22, S23, S25, S26, S29, S30, S292, S311, S318, S343, U.S. Department of Health, Education, and Welfare; Publ. (NIOSH) 77-185 (1977).

METHOD REVISED BY: R. Alan Lunsford, Ph.D., and Julie R. Okenfuss; based on results of NIOSH Contract CDC-99-74-45.

Table 1. Synonyms, formula, molecular weight, properties [5].

Name/Synonyms	Structure	Empirical Formula	Molec- ular Weight	Boiling Point (°C)	Vapor Pressure @ 25 °C		Density @ 20 °C (g/mL)
					(mm Hg)	(kPa)	
benzene CAS #71-43-2		C <sub>6</sub> H <sub>6</sub>	78.11	80.1	95.2	12.7	0.879
p-tert-butyltoluene CAS #98-51-1 1-tert-butyl-4-methylbenzene		C <sub>11</sub> H <sub>16</sub>	148.25	192.8	0.7	0.09	0.861
cumene CAS #98-82-8 isopropylbenzene		C <sub>9</sub> H <sub>12</sub>	120.20	152.4	4.7	0.62	0.862
ethylbenzene CAS #100-41-4		C <sub>8</sub> H <sub>10</sub>	106.17	136.2	9.6	1.28	0.867
α-methylstyrene CAS #98-83-9 isopropenylbenzene (1-methylethenyl)-benzene		C <sub>9</sub> H <sub>10</sub>	118.18	165.4	2.5	0.33	0.911
naphthalene CAS #91-20-3		C <sub>10</sub> H <sub>8</sub>	128.18	80.2 <sup>a</sup>	0.2	0.03	1.025
styrene CAS #100-42-5 vinylbenzene		C <sub>8</sub> H <sub>8</sub>	104.15	145.2	6.1	0.81	0.906
toluene CAS #108-88-3 methylbenzene		C <sub>7</sub> H <sub>8</sub>	92.14	110.6	28.4	3.79	0.867
vinyltoluene <sup>b</sup> CAS #25013-15-4 methylstyrene methylvinylbenzene		C <sub>9</sub> H <sub>10</sub>	118.18	167.7	1.6	0.22	0.898
				(meta) 171.6	1.9	0.26	0.911
				(para) 172.8	1.8	0.24	0.911
				(ortho) 169.8	1.8	0.24	0.904
xylene <sup>c</sup> CAS #1330-20-7 dimethylbenzene		C <sub>8</sub> H <sub>10</sub>	106.17	(ortho) 144.4	6.7	0.89	0.880
				(meta) 139.1	8.4	1.12	0.864
				(para) 138.4	8.8	1.18	0.861

<sup>a</sup>Melting point.<sup>b</sup>Commercial mixture of meta and para isomers.<sup>c</sup>Mixture of isomers.

Table 2. Permissible exposure limits, ppm [6-11].

Substance	OSHA			NIOSH		ACGIH		mg/m <sup>3</sup> per ppm
	TWA	C	Peak	TWA	C	TLV	STEL	
benzene	10	25	50 <sup>a</sup>	1		10**	25**	3.19
p-tert-butyltoluene	10					10	20	6.06
cumene	50 (skin)					50	75 (skin)	4.91
ethylbenzene	100					100	125	4.34
α-methylstyrene		100				50	100	4.83
naphthalene	10					10	15	5.24
styrene	100	200	600 <sup>b</sup>	50	100	50	100	4.26
toluene	200	300	500 <sup>a</sup>	100	200*	100	150 (skin)	3.77
vinyltoluene	100					50	100	4.83
xylene	100			100	200*	100	150	4.34

<sup>a</sup>Maximum duration 10 min in 8 hr.

\*\*ACGIH: suspect carcinogen [10].

<sup>b</sup>Maximum duration 5 min in any 3 hr.

\* 10-min sample.

Table 3. Sampling flowrate<sup>a</sup>, volume, capacity, range, overall bias and precision [3,4,12].

Substance	Sampling			Breakthrough		Range	Overall	
	Flowrate (L/min)	Volume (L) VOL-NOM	VOL-MAX <sup>b</sup>	Volume @ Concentration (L) (mg/m <sup>3</sup> )		at VOL-NOM (mg/m <sup>3</sup> )	Bias (%)	Precision (s <sub>r</sub> )
benzene	≤0.20	2 <sup>c</sup>	30	>45	149	42- 165	0.8	0.059
p-tert-butyltoluene	≤0.20	10	29	44	112	29- 119	-10.4	0.071 <sup>d</sup>
cumene	≤0.20	10	30	>45	480	120- 480	4.6	0.059
ethylbenzene	≤0.20	10	24	35	917	222- 884	-8.1	0.089 <sup>d</sup>
α-methylstyrene	≤0.20	3 <sup>f</sup>	30	>45	940	236- 943	-10.8	0.061 <sup>d</sup>
naphthalene <sup>e</sup>	≤1.0	200	200	>240	81	19- 83	-0.5	0.055
styrene	≤1.0	59	14	21	1710	426-1710	-10.7	0.058 <sup>d</sup>
toluene	≤0.20	26	8	12	2294	548-2190	3.8	0.052
vinyltoluene	≤0.20	10	24	36	952	256- 970	-9.5	0.061 <sup>d</sup>
xylene	≤0.20	12	23	35	870	218- 870	-2.1	0.060

<sup>a</sup>Minimum recommended flow is 0.01 L/min.<sup>b</sup>Approximately two-thirds the breakthrough volume, except for naphthalene.<sup>c</sup>10-min sample.<sup>d</sup>Corrected value, calculated from data in Reference 12.<sup>e</sup>Naphthalene shows poor desorption efficiency at low loading; 100-L minimum volume is recommended.<sup>f</sup>15-min sample.

95-min sample.

Table 4. Measurement range, precision and conditions<sup>a</sup> [3,4,12].

Substance	Desorption Volume (mL)	Measurement		Carrier Flow (mL/min)	Column Parameters <sup>b</sup>		
		Range (mg)	Precision (s <sub>r</sub> )		t <sub>r</sub> - Length (°C)	(m)	Packing <sup>c</sup>
benzene	1.0	0.09- 0.35	0.036	50	115	0.9	A
p-tert-butyltoluene	0.5	0.27- 1.09	0.021 <sup>d</sup>	50	115	3.0	B
cumene	0.5	0.86- 3.46	0.010	50	99	3.0	B
ethylbenzene	0.5	2.17- 8.67	0.010	50	85	3.0	B
α-methylstyrene	0.5	0.69- 3.57	0.011	50	115	3.0	B
naphthalene	1.0	4.96-19.7	0.019	30	125	3.0	C
styrene	0.5	2.17- 8.49	0.013 <sup>d</sup>	50	109	3.0	B
toluene	1.0	1.13- 4.51	0.011	50	155	0.9	D
vinyltoluene	0.5	2.41- 9.64	0.008	50	120	3.0	B
xylene	1.0	2.60-10.4	0.010	50	180	0.9	D

<sup>a</sup>Injection volume, 5.0 µL; nitrogen carrier gas.<sup>b</sup>All columns stainless steel, 3.2 mm outside diameter.<sup>c</sup>A, 50/80 mesh Porapak P; B, 10% FFAP on 80/100 mesh Chromosorb W AW-DMCS;

C, 10% OV-101 on 100/120 mesh Supelcoport; D, 50/80 mesh Porapak Q.

<sup>d</sup>Corrected value, calculated from data in [12].

FORMULA: Table 1

HYDROCARBONS, HALOGENATED

M.W.: Table 1

METHOD: 1003

ISSUED: 2/15/84

REVISION #1: 8/15/87

COMPOUNDS: benzyl chloride	chlorobromomethane	1,1-dichloroethane	methylchloroform
(synonyms bromoform	chloroform	1,2-dichloroethylene	tetrachloroethylene
in Table 1) carbon tetrachloride	o-dichlorobenzene	ethylene dichloride	1,1,2-trichloroethane
chlorobenzene	p-dichlorobenzene	hexachloroethane	1,2,3-trichloropropane

SAMPLING	MEASUREMENT
SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	! TECHNIQUE: GAS CHROMATOGRAPHY, FID
FLOW RATE: 0.01 to 0.2 L/min	! ANALYTE: compounds above
VOL-MIN: Table 2	! DESORPTION: 1 mL CS <sub>2</sub> , stand 30 min
-MAX: Table 2	! INJECTION VOLUME: 5 µL
SHIPMENT: routine	! TEMPERATURES: Table 3
SAMPLE STABILITY: not determined	! CARRIER GAS: N <sub>2</sub> or He, 30 mL/min
FIELD BLANKS: 10% of samples	! COLUMN: Table 3; alternates are SP-2100, SP-2100 with 0.1% Carbowax 1500 or DB-1 fused silica capillary column
ACCURACY	! CALIBRATION: standard solutions of analyte in CS <sub>2</sub>
RANGE STUDIED: see EVALUATION OF METHOD [1]	! RANGE: Table 3
BIAS: not significant [1]	! ESTIMATED LOD: 0.01 mg per sample [2]
OVERALL PRECISION (s <sub>p</sub> ): see EVALUATION OF METHOD [1]	! PRECISION (s <sub>p</sub> ): see EVALUATION OF METHOD [1]

APPLICABILITY: See Table 2 for working ranges. This method can be used for simultaneous determination of two or more substances suspected to be present by changing gas chromatographic conditions (i.e., temperature program). High humidity during sampling will prevent organic vapors from being trapped efficiently on the sorbent and greatly decreases breakthrough volume.

INTERFERENCES: ~~None identified.~~ The chromatographic column or separation conditions may be changed to ~~circumvent~~ interferences.

OTHER METHODS: ~~This method combines and replaces P&CAM 127 [3], S101 [4], S110 [5], S113 [6], S114 [7], S115 [8], S122 [9], S123 [10], S126 [11], S133 [12], S134 [13], S135 [14], S281 [15], S314 [16], S328 [17], S335 [18], S351 [19], and Method 1003 (dated 2/15/84).~~

## REAGENTS:

1. Carbon disulfide, chromatographic quality.\*
2. Analyte, reagent grade.
3. Calibration stock solutions:
  - a. benzyl chloride, 10 mg/mL in *n*-heptane.
  - b. bromoform, 10 mg/mL in *n*-hexane.
  - c. *o*-dichlorobenzene, 200 mg/mL in acetone.
  - d. *p*-dichlorobenzene, 300 mg/mL in acetone.
  - e. hexachloroethane, 25 mg/mL in toluene.
4. Decane, *n*-undecane, octane or other internal standards (see step 6).
5. Nitrogen or helium, purified.
6. Hydrogen, prepurified.
7. Air, filtered.

## EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6 mm OD, 4 mm ID, flame-sealed ends with plastic caps, containing two sections of 20/40 mesh activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available (e.g., SKC #226-01).
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Gas chromatograph, FID, integrator and column (see Table 3).
4. Vials, 2-mL, glass, PTFE-lined septum crimp caps.
5. Volumetric flasks, 10-mL.
6. Syringes, 10- $\mu$ L, readable to 0.1  $\mu$ L.
7. Pipet, TD, 1-mL, with pipet bulb.

\*See SPECIAL PRECAUTIONS.

**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C); work with it only in a hood. Several of the analytes are suspect carcinogens (Table 1). *n*-Heptane, *n*-hexane, and acetone are fire hazards.

## SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size between the limits shown in Table 2.
4. Cap the samplers. Pack securely for shipment.

## SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL CS<sub>2</sub> to each vial. Cap each vial.

NOTE: A suitable internal standard, such as decane [16], *n*-undecane [6,19], or octane [9,13,17] at 0.1% (v/v) may be added at this step and at step 8.

7. Allow to stand 30 min with occasional agitation.

## CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least five working standards over the appropriate range (Table 3).
  - a. Add known amounts of neat analyte or calibration stock solution to CS<sub>2</sub> in 10-mL volumetric flasks and dilute to the mark.
  - b. Analyze with samples and blanks (steps 11 and 12).
  - c. Prepare calibration graph (peak area vs. mg analyte).
9. Determine desorption efficiency (DE) at least once for each lot of charcoal used for sampling in the range of interest. Prepare three tubes at each of five levels plus three media blanks.



- a. Remove and discard back sorbent section of a media blank sampler.
  - b. Inject a known amount (2 to 20  $\mu\text{L}$ ) of pure analyte, or calibration stock solution (see REAGENTS, 3.), directly onto front sorbent section with a microliter syringe.
  - c. Cap the tube. Allow to stand overnight.
  - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
  - e. Prepare a graph of DE vs. mg analyte recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

## MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1003-1 and in Table 3. Inject sample aliquot manually using solvent flush technique or with autosampler.

NOTE: If peak area is above the linear range of the working standards, dilute with  $\text{CS}_2$ , reanalyze and apply the appropriate dilution factor in calculations.

12. Measure peak area.

## CALCULATIONS:

13. Determine the mass, mg (corrected for DE), of analyte found in the sample front ( $W_f$ ) and back ( $W_b$ ) sorbent sections and in the average media blank front ( $B_f$ ) and back ( $B_b$ ) sorbent sections.

NOTE: If  $W_b > W_f/10$ , report breakthrough and possible sample loss.

14. Calculate concentration, C, of analyte in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \text{ mg/m}^3.$$

## EVALUATION OF METHOD:

Laboratory testing was performed with spiked samples and generated atmospheres using SKC Lot 105 coconut shell charcoal [1]. Results were:

Compound	Range, mg/m <sup>3</sup>	Sample Size	Precision ( $s_p$ )		Desorption Efficiency	Ref.
			Overall	Measurement		
Benzyl chloride	2-8	10 L	0.096	0.031	0.90 @ 0.03-0.1 mg	[8]
Bromoform	3-10	10 L	0.071	0.043	0.80 @ 0.025 mg	[7]
Carbon tetrachloride	65-299	15 L	0.092	0.037	0.96 @ 1.3-4.8 mg	[16]
Chlorobenzene	183-736	10 L	0.056	0.025	0.91 @ 1.8-7.1 mg	[12]
Chlorobromomethane	640-2655	5 L	0.061	0.051	0.94 @ 3.3-13 mg	[6]
Chloroform	100-416	15 L	0.057	0.047	0.97 @ 1.8-7.4 mg	[19]
<i>o</i> -Dichlorobenzene	150-629	3 L	0.068	0.013	0.86 @ 0.5-1.9 mg	[14]
<i>p</i> -Dichlorobenzene	183-777	3 L	0.052	0.022	0.91 @ 0.7-2.7 mg	[15]
1,1-Dichloroethane	212-838	10 L	0.057	0.011	1.01 @ 1.9-8 mg	[10]
1,2-Dichloroethylene*	475-1915	3 L	0.052	0.017	1.00 @ 2.4-9.5 mg	[5]
Ethylene dichloride	195-819	3 L	0.079	0.012	0.96 @ 0.6-2.5 mg	[9]
Hexachloroethane	5-25	10 L	0.121	0.014	0.98 @ 0.05-0.2 mg	[4]
Methyl chloroform	904-3790	3 L	0.054	0.018	0.99 @ 2.9-11 mg	[17]
Tetrachloroethylene	655-2749	3 L	0.052	0.013	0.96 @ 2.1-8 mg	[18]
1,1,2-Trichloroethane	26-111	10 L	0.057	0.010	0.97 @ 0.3-1.2 mg	[13]
1,2,3-Trichloropropane	163-629	10 L	0.068	0.027	0.95 @ 1.5-6 mg	[11]

\*isomer used (i.e., cis- or trans-) in evaluation unknown.

## REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S101, S110, S113, S114, S115, S122, S123, S126, S133, S134, S135, S281, S314, S328, S335, S351, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.
- [2] User check, UBTI, NIOSH Sequences #3990-T, 3990-U and 3990-W (NIOSH, unpublished, November 3, 1983) and 4304-J (NIOSH, unpublished, April 3, 1984).
- [3] NIOSH Manual of Analytical Methods, 2nd ed., V. 1., P&CAM 127, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [4] Ibid., V. 2., S101, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [5] Ibid., S110.
- [6] Ibid., S113.
- [7] Ibid., S114.
- [8] Ibid., S115.
- [9] Ibid., S122.
- [10] Ibid., S123.
- [11] Ibid., S126.
- [12] Ibid., S133.
- [13] Ibid., S134.
- [14] Ibid., V. 3, S135, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [15] Ibid., S281.
- [16] Ibid., S314.
- [17] Ibid., S328.
- [18] Ibid., S335.
- [19] Ibid., S351.
- [20] NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards, U.S. Department of Health and Human Services, Publ. (NIOSH) 81-123 (1981), available as Stock #PB83-154609 from NTIS, Springfield, VA 22161.
- [21] NIOSH Current Intelligence Bulletin 27, Chloroethanes: Review of Toxicity, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-181 (1978).
- [22] NIOSH Current Intelligence Bulletin 20, Tetrachloroethylene (Perchloroethylene), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-112 (1978).
- [23] Criteria for a Recommended Standard...Occupational Exposure to Benzyl Chloride, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-182 (1978).
- [24] Criteria for a Recommended Standard...Occupational Exposure to Carbon Tetrachloride, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-133 (1976).
- [25] Criteria for a Recommended Standard...Occupational Exposure to Chloroform, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 75-114 (1975).
- [26] Criteria for a Recommended Standard...Occupational Exposure to Ethylene Dichloride, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-139 (1976).
- [27] Criteria for a Recommended Standard...Occupational Exposure to 1,1,1-Trichloroethane, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-184 (1976).
- [28] Criteria for a Recommended Standard...Occupational Exposure to Tetrachloroethylene (Perchloroethylene), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-185 (1976).

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Table 1. General information.

Compound	M.W.	mg/m <sup>3</sup> = 1 ppm @ NTP	Synonyms	OSHA/NIOSH/ACGIH (ppm)
Benzyl chloride* (C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl)	126.58	5.17	(chloromethyl) benzene; α-chlorotoluene; CAS #100-44-7	1/—/1 [20,23]
Bromoform (CHBr <sub>3</sub> )	252.75	10.33	tribromomethane; CAS #75-25-2	0.5/—/0.5 (skin) [20]
Carbon tetrachloride* (CCl <sub>4</sub> )	153.84	6.29	tetrachloromethane; CAS #56-23-5	10, C 25/C 2/5 (skin) [20,24]
Chlorobenzene (C <sub>6</sub> H <sub>5</sub> Cl)	112.56	4.60	monochlorobenzene; phenyl chloride; CAS #108-90-7	75/—/75 [20]
Chlorobromomethane (CH <sub>2</sub> BrCl)	129.39	5.29	bromochloromethane; Halon 1011; CAS #74-97-5	200/—/200, STEL 250 [20]
Chloroform* (CHCl <sub>3</sub> )	119.39	4.88	trichloromethane; CAS #67-66-3	C 50/C 2/10 [20,25]
o-Dichlorobenzene (1,2-C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub> )	147.00	6.01	1,2-dichlorobenzene; CAS #95-50-1	50/—/C 50 [20]
p-Dichlorobenzene (1,4-C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub> )	147.00	6.01	1,4-dichlorobenzene; CAS #106-46-7	75/—/75, STEL 110 [20]
1,1-Dichloroethane (CH <sub>3</sub> CHCl <sub>2</sub> )	98.96	4.05	ethylidene chloride; CAS #75-34-3	100/100/200, STEL 250 [20,21]
1,2-Dichloroethylene (ClCH=CHCl)	96.94	3.96	acetylene dichloride; 1,2-dichloroethene; CAS #540-59-0	200/—/200, STEL 250 [20]
Ethylene dichloride* (ClCH <sub>2</sub> CH <sub>2</sub> Cl)	98.96	4.05	1,2-dichloroethane; CAS #107-06-2	50, C 100/5, C 15/10 [20,21,26]
Hexachloroethane* (CCl <sub>3</sub> CCl <sub>3</sub> )	236.74	9.68	perchloroethane; CAS #67-72-1	1 (skin)/—/10 [20,21]
Methylchloroform (CH <sub>3</sub> CCl <sub>3</sub> )	133.42	5.45	1,1,1-trichloroethane; CAS #71-55-6	350/C 350/350, STEL 450 [20,21,27]
Tetrachloroethylene (Cl <sub>2</sub> C=CCl <sub>2</sub> )	165.83	6.78	perchloroethylene; CAS #127-18-4	100, C 200, P 300/—/ 50, STEL 200 [20,28]
1,1,2-Trichloroethane (Cl <sub>2</sub> CHCH <sub>2</sub> Cl)	133.41	5.45	vinyl trichloride; CAS #79-00-5	10 (skin)/—/10 (skin) [20,21]
1,2,3-Trichloropropane (CH <sub>2</sub> ClCHClCH <sub>2</sub> Cl)	147.43	6.03	allyl trichloride; glycerol trichlorohydrin; CAS #96-18-4	50/—/50, STEL 75 [20]

\*Suspect carcinogen [20,21,22 ].

Table 2. Sampling limits.

Compound	Air Sample Volume (L)			Working Range, ppm, at Max Sample Volume
	Min	Max	Target	
Benzyl chloride	6 @ 1 ppm	50	10	0.6 to 5.8
Bromoform	4 @ 0.5 ppm	70	10	0.2 to 4
Carbon tetrachloride	3 @ 10 ppm	150	15	2 to 105
Chlorobenzene	1.5 @ 75 ppm	40	10	10 to 430
Chlorobromomethane	0.5 @ 200 ppm	8	5	18 to 450
Chloroform	1 @ 50 ppm	50	15	2 to 190
<i>o</i> -Dichlorobenzene	1 @ 50 ppm	60	3	16 to 1100
<i>p</i> -Dichlorobenzene	1 @ 75 ppm	10	3	27 to 330
1,1-Dichloroethane	0.5 @ 100 ppm	15	10	4 to 250
1,2-Dichloroethylene	0.2 @ 200 ppm	5	3	16 to 560
Ethylene dichloride	1 @ 50 ppm	50	3	16 to 1320
Hexachloroethane	3 @ 1 ppm	70	10	0.3 to 8.3
Methylchloroform	0.1 @ 350 ppm	8	3	18 to 1450
Tetrachloroethylene	0.2 @ 100 ppm	40	3	9 to 1900
1,1,2-Trichloroethane	2 @ 10 ppm	60	10	1.8 to 64
1,2,3-Trichloropropane	0.6 @ 50 ppm	60	10	3 to 310

Table 3. Measurement parameters.

Compound	Column*	t (°C)	Range (mg per sample)
		Column/Injector/Detector	
Benzyl chloride	A	160/170/210	0.02 to 0.15
Bromoform	A	130/170/210	0.02 to 0.15
Carbon tetrachloride	B	60/155/200	0.2 to 7
Chlorobenzene	A	105/190/250	0.4 to 10
Chlorobromomethane	A	80/170/210	0.5 to 15
Chloroform	B	75/155/200	0.4 to 11
<i>o</i> -Dichlorobenzene	C	140/225/250	0.1 to 3
<i>p</i> -Dichlorobenzene	A	140/225/275	0.2 to 4
1,1-Dichloroethane	A	50/100/175	0.4 to 12
1,2-Dichloroethylene	A	60/170/210	0.2 to 7
Ethylene dichloride	C	70/225/250	0.1 to 4
Hexachloroethane	D	110/170/210	0.02 to 0.3
Methylchloroform	C	70/225/250	0.6 to 17
Tetrachloroethylene	C	90/225/250	0.4 to 12
1,1,2-Trichloroethane	C	70/250/225	0.05 to 2
1,2,3-Trichloropropane	E	160/180/230	0.3 to 9

\*A = 3 m x 3-mm OD stainless steel, 10% SP-1000 on 80/100 mesh Chromosorb WHP.

B = 6 m x 3-mm OD, otherwise same as A.

C = 3 m x 3-mm OD stainless steel, 10% OV-101 on 100/120 mesh Chromosorb WHP.

D = 3 m x 6-mm OD glass, 3% SP-2250 on 80/100 mesh Chromosorb WHP.

E = 3 m x 3-mm OD stainless steel, 10% FFAP on 80/100 mesh Chromosorb WHP.